PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: G. Vilcins
PERIOD COVERED: January, 1989

I. THERMOGRAVIMETRIC ANALYSIS (TGA)

A. **Objective**: To analyze carbon and low side stream smoke cigarette paper by TGA.

B. **Results**:

Four new carbon samples with high iron content, Vantage Excel Cigarette paper and related papers were analyzed using TGA.

Two of the new carbon samples containing 60 percent and 70 percent iron content actually gained weight due to the oxidation of the iron. The predominant reaction in the samples containing 20 percent and 30 percent iron was combustion.

Low side stream smoke cigarette papers with known compositions were used to develop a method for the determination of magnesium hydroxide and calcium carbonate content in the Vantage Excel cigarette paper. Based on this method the concentrations of magnesium hydroxide and calcium carbonate in the Vantage Exel cigarette paper were 24 and 17 percent, respectively.

C. **Conclusions**: By decomposing all or nearly all the cellulose in low side stream smoke cigarette paper isothermally at a temperature which will not decompose magnesium hydroxide, quantitative analysis can be achieved for it in any low side stream smoke cigarette paper.

D. **Plans**: The work is still in progress.

E. **References**:

Chung, C., PM Notebook 8752, pp. 26-48.

II. PYROLYSIS-GC/MS OF MENTHOL RELEASE POLYMER

A. **Objective**: To examine the release of menthol at a range of temperatures under Curie-Point Pyrolysis conditions.

B. **Results**: Polymic was pyrolyzed at 9 temperatures ranging from 160°C to 920°C. The identities of the volatile gases produced were determined by GC/MS, and estimates of the amount of menthol and menthenes released were determined.

C. **Conclusions**: Conclusions are still being drawn, although preliminary data indicate that menthol release increases with increasing temperature up to ~300°C, where the release levels off until it begins to drop slowly at ~650°C. Menthenes begin to

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appear in significant quantities at ~250°C, and then gradually level off between ~450° and ~650°C. The release of menthenes maximizes at ~750°C, and then drops again. The ratio of menthol to menthenes peaks at the 268°C pyrolysis temperature, although the amount of menthol released at that temperature is only ~60% of the maximum plateau amount of menthol released (at pyrolysis temperatures of~350°C to 600°C).

D. **Plans**: A memo will be written reporting details of the study.

E. **References**:

Magin, D., PM Notebook 8534, page 200.

III. SMOKE ANALYSIS

A. **Objective**: The effect of DIET expansion on the smoke chemistry of ART filler.

B. **Results**: The 100% ART-DIET cigarette, compared with the 100% ART FEED cigarette, delivered to mainstream smoke more pyrazines, phenolics, sugar-related degradation products, isovaleric, and 2-methylvaleric acids; less volatile pyridines, beta-methylvaleric, and 2-methylbutyric acids. The expanded filler delivered comparable amounts of higher fatty acids, simple aromatics, aromatic acids, and tobacco essential oil-related products. The ART-DIET filler shared some characteristics of ET: high in ammonia and low in total reducing sugar. The occurrence of browning reactions prior to and during smoking is more evident than in the previous study.

C. **References**:

Hsu, F. and A. Oti, "Smoke Chemistry - ART/DIET," memo to B. Handy, January 20, 1989.

IV. MASS SPECTROMETRY OF LIPID MATERIALS

A. **Objective**: To develop methodology, utilizing soft ionization and tandem mass spectrometry, for characterization of complex lipid mixtures.

B. **Results**: Work with lipid extracts from tobacco before and after ART processing, samples of residue from the ART process, and known lipid mixtures continues. Evaluation of data obtained from analyses performed using the MS-50 triple analyzer mass spectrometer at the Midwest Center for Mass Spectrometry has revealed much useful information. However, considering the exploratory and developmental nature of the experiments more analyses will be required to confirm and explore potentially significant observations.

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C. **Plans**: Experiments are being designed to explore potentially significant data.

D. **References**:

Jensen, N., PM Notebook 8620, pp.44 -64.

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